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(54) Title: PERFUMED COLOURED SPECKLE COMPOSITION AND PARTICULATE LAUNDRY DETERGENT COMPOSITIONS CONTAINING IT

(57) Abstract: A perfumed speckle composition which comprises at least 1 wt% perfume, preferably at least 1.5 wt% and is preferably made by a process comprising the steps of (i) mixing an aqueous perfume emulsion and a colourant with an inorganic granular carrier material and (ii) layering the resultant material with a finely divided porous particulate material. The invention also relates to detergent compositions comprising a minor proportion of a coloured speckle composition wherein at least 10 wt%, preferably at least 20 wt%, more preferably at least 30 wt%, of the total amount of perfume in the detergent composition is located in the speckles.

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PERFUMED COLOURED SPECKLE COMPOSITION AND PARTICULATE LAUNDRY DETERGENT COMPOSITIONS CONTAINING IT

TECHNICAL FIELD

The present invention relates to coloured perfumed speckles for use in granular laundry detergent compositions and a process for making them.

BACKGROUND AND PRIOR ART

Commercial laundry detergent washing powders often contain ingredients which do not provide any cleaning function but provide an additional benefit to the consumer. One such commonly used ingredient is perfume. Perfume is added to improve aesthetic appeal by imparting a pleasing odour both during storage and in use. Additionally it may impart a pleasing odour to the washed fabrics. By their very nature perfumes are highly volatile and the amount remaining on the washed clothes by the end of the washing process is usually a small fraction of that which was added at the beginning.

There have been many attempts to improve and control the release of perfume during the wash process.

WO 9734981 (Procter & Gamble) discloses a porous zeolite into which the perfume is absorbed and trapped by a release inhibitor which hydrolyses during the wash thereby releasing the perfume in a controlled manner.

WO 9842818 (Procter & Gamble) discloses a porous core particle encapsulated in a glassy material and coated in a water-soluble or dispersible compound. Preferably a perfume is supported or contained within the porous carrier.

EP 466235 (Quest) discloses a process of combining a perfume with one or more non-ionic emulsifiers to form a non-aqueous phase which is then mixed with an aqueous phase to form a structured emulsion containing liquid-crystal structures. The emulsified perfume is added to a detergent powder and is said to enhance perfume delivery and possibly deposition.

Another ingredient which is commonly added and yet provides little or no washing function is visually striking 'speckles'. Their primary function is to provide an attractive colouration to the powder and a cue to prompt the consumer into associating the speckles with a separate functional effect.

US 5 605 883 (Iliff et al) discloses a colourant speckle which exhibits reduced spotting of teated fabrics. It also states that the speckle may be used as a vehicle for carrying cosurfactants, enzymes, oxidants, bleach activators and fragrances.

US 4 097 418 (Procter & Gamble) discloses a granular coloured speckle having reduced staining properties and may comprise up to 3% of enzymes, bleaching agents, antimicrobial agents, corrosion inhibitors and perfume.

SUMMARY OF INVENTION

Surprisingly the present inventors have discovered that the delivery of perfume may be significantly enhanced and prolonged if the perfume is included in coloured speckles. Especially advantageous perfume delivery can be achieved if the perfume is incorporated in the speckles in aqueous emulsion form. Additionally the colouration of the speckles may provide an association with the enhanced perfume delivery in the mind of the consumer, thus reinforcing the consumer's appreciation of the wash process.

STATEMENT OF INVENTION

In a first aspect, the present invention provides a perfumed coloured granular composition for use as speckles in a particulate laundry detergent composition which comprises at least 1 wt% perfume.

In a second aspect, the present invention provides a process for the manufacture of a perfumed coloured granular composition for use as speckles in a particulate laundry detergent composition, comprising the steps of:

(i) mixing an aqueous perfume emulsion and a colourant with an inorganic granular carrier material; and
(ii) layering the resultant material with a finely divided porous particulate material.

In a third aspect, the present invention provides a perfumed particulate laundry detergent composition comprising a minor

proportion of a coloured speckle composition wherein at least 10 wt% of the total amount of perfume in the detergent composition is located in the speckles.

DETAILED DESCRIPTION OF THE INVENTION

The Speckles

The speckles (perfumed coloured granular composition) comprise a perfume, preferably in aqueous emulsion form, and a colourant, on an inorganic granular carrier material. The speckles comprise at least 1 wt% perfume and preferably comprise at least 1.5 wt%.

It is preferred that, before drying, the perfume is present in the form of an aqueous emulsion. In this case the speckles suitably comprise emulsifier. It is preferred that the speckles are obtainable from a process as defined in detail below.

The Perfume

As used herein the term "perfume" denotes one or a mixture of perfume components, optionally mixed with a suitable solvent, diluent or carrier, which is used to impart a desired odour to the particulate laundry detergent composition in the package and/or in use and/or to the fabrics that are washed.

A range of perfumes and solvents which may be used are disclosed in EP 466 235 B (Quest).

The Aqueous Emulsion

The perfume is preferably incorporated in the speckles as an aqueous emulsion. Such emulsions suitably contain from 1 to 50 wt% of perfume, from 1 to 30 wt% of one or more non-ionic emulsifiers and from 20 to 98 wt% of water or an aqueous mixture containing water-soluble and/or water-dispersible materials, hereinafter jointly referred to as "aqueous phase". Such water-soluble or water-dispersible materials may form up to 30 wt% of the aqueous phase.
It is particularly suitable that the weight ratio of total emulsifier to perfume lies within the range of from 1:5 to 3:1 and the weight ratio of non-aqueous phase to aqueous phase lies within the range of from 1:2 to 4:3, preferably within the range of from 1:2 to 1:1.

Non-ionic emulsifiers suitable for use in the present invention are exemplified in EP 466 235 B, for example the primary and secondary alcohol ethoxylates, especially the C_8-C_{20} aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C_{10}-C_{15} primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol, preferably from 4 to 9. Non-ethoxylated nonionic surfactants include alkylpolyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

An especially preferred nonionic emulsifier is C_{10}-C_{15}, preferably C_{12}-C_{15}, aliphatic alcohol ethoxylated with an average of from 4 to 9 moles of ethylene oxide, preferably 5 to 7, per mole of alcohol.

The emulsion may be produced by forming a non-aqueous phase comprising the perfume, a non-ionic emulsifier or mixture based on non-ionic emulsifiers, and optionally other adjuncts, which is mixed at a temperature at which the non-aqueous phase forms a homogeneous liquid; forming an aqueous phase consisting of water or an aqueous solution of water-soluble or water-dispersible materials; and finally mixing the two phases under shear conditions.

The Process

The perfumed coloured granular composition for use as speckles in a particulate laundry detergent composition according to the present invention may be manufactured by the following process:

(i) mixing an aqueous perfume emulsion and a colourant with an inorganic granular carrier material; and
(ii) layering the resultant material with a finely divided porous particulate material.

The colourant may be a pigment or a water-dispersible dye.

Optionally the inorganic carrier material may be layered prior to step (i) with a finely divided porous particulate

material. This step may be particularly advantageous when the inorganic carrier material has a low liquid carrying capacity, for example sodium chloride.

The speckle composition may be prepared using any suitable mixing apparatus. The mixer should preferably have a stainless steel or other inert interior surface especially when a corrosive carrier material, for example, sodium chloride, is used.

The granular carrier material may be any suitable material compatible with granular detergent compositions, such as for example hydratable alkaline inorganic salts such as sodium tripolyphosphate, sodium carbonate or sodium tetraborate; solid surfactant e.g. highly ethoxylated nonionic surfactant; alpha-hydroxy carboxylic acids e.g. citric acid; or other materials such as sodium chloride, crystalline alkali metal aluminosilicate, sodium sulphate, soap, sodium metasilicate, clays and corn starch. Sodium chloride is especially preferred.

The granular carrier material should have a particle size similar to that of a typical detergent powder. Typically it has a number average particle size of at least 100 microns.

Once the emulsified perfume has been mixed with the granular carrier material, a porous particulate layering material is then used to provide a dry layer to protect the emulsified perfume and to provide a flowable powder. The layering material may suitably comprise at least one highly absorbent

material selected from silicas, silicates and crystalline alkali metal aluminosilicates.

The porous particulate layering material advantageously comprises a material having an oil absorption capacity of at least 0.4 ml/g.

Advantageously the porous particulate layering material has a number average particle size of at most 100 microns.

The total amount of layering agent is suitably from 0.1 to 10 wt%, preferably from 0.2 to 1 wt% based on the speckles.

Optionally the speckle composition may also contain a binder. Preferred binders are polymers, for example, cellulosic materials, for example, sodium carboxymethyl cellulose, or acrylic polymers, for example, acrylic/maleic copolymers such as Sokalan (Trade Mark) CP5 ex BASF. Other organic film-forming materials may be envisaged, for example, polyethylene glycols or highly ethoxylated nonionic surfactants. An especially preferred binder is sodium carboxymethyl cellulose. The binder is suitably present in an amount of from 0.05 to 5 wt%, preferably from 0.1 to 1 wt%.

The Perfumed Particulate Laundry Detergent Composition

The coloured speckle composition of the invention is suitable for use in both white and coloured detergent compositions, provided that there is sufficient contrast

between the colour of the major proportion of the powder and the colour of the speckles.

Thus the detergent composition may comprise a major proportion of white or light-coloured particles and a minor proportion of the coloured speckle composition. Alternatively, the detergent composition may comprise a major proportion of particles having a first colour, for example, blue, and a minor proportion of the coloured speckle composition having a second colour contrasting with the first colour, for example, red, or having a darker shade of the same colour.

The speckle composition is preferably present in an amount of from 1 to 10 wt% and preferably from 2 to 8 wt%.

In a preferred embodiment, the detergent composition of the present invention comprises a minor proportion of a coloured speckle composition wherein at least 10 wt% of the total amount of perfume in the detergent composition is located in the speckles. Preferably at least 20 wt%, more preferably at least 30 wt% of the perfume is located in the speckles. Any remaining perfume may be incorporated in the bulk powder by any suitable method. It is believed that this may provide a particularly satisfactory perfume release profile. It is preferred that the detergent composition of the present invention comprises a speckle composition as described above and/or a speckle composition obtainable by a process as described above.

The detergent compositions of the invention may be in powder or tablet form.

Detergent Ingredients

Detergent compositions according to the invention contain, as well as the speckle composition, conventional detergent ingredients, notably detergent-active materials (surfactants), and preferably also detergency builders.

Laundry detergent compositions in accordance with the invention may suitably comprise from 5 to 60 wt% of detergent-active surfactant, from 10 to 80 wt% of detergency builder, from 1 to 10 wt% of the speckles, and optionally other detergent ingredients to 100 wt%.

The detergent compositions will contain, as essential ingredients, one or more detergent active compounds (surfactants) which may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent active compounds, and mixtures thereof. Many suitable detergent active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

The preferred detergent active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds. Non-soap anionic surfactants are especially preferred.

Non-soap anionic surfactants are well-known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl chain length of C_8-C_{15}; primary and secondary alkylsulphates, particularly C_8-C_{15} primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred. A preferred anionic surfactant is linear alkylbenzene sulphonate.

Nonionic surfactants may optionally be present. These include the primary and secondary alcohol ethoxylates, especially the C_8-C_{20} aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C_{10}-C_{15} primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpoly-glycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

Cationic surfactants may optionally be present. These include quaternary ammonium salts of the general formula $R_1R_2R_3R_4N^+ X^-$ wherein the R groups are long or short hydrocarbyl chains, typically alkyl, hydroxyalkyl or ethoxylated alkyl groups, and X is a solubilising anion (for example, compounds in which R_1 is a C_8-C_{22} alkyl group, preferably a C_8-C_{10} or C_{12}-C_{14} alkyl group, R_2 is a methyl

group, and R_3 and R_4, which may be the same or different, are methyl or hydroxyethyl groups); and cationic esters (for example, choline esters).

In an especially preferred cationic surfactant of the general formula $R_1R_2R_3R_4N^+ X^-$, R_1 represents a C_8-C_{10} or C_{12}-C_{14} alkyl group, R_2 and R_3 represent methyl groups, R_4 presents a hydroxyethyl group, and X^- represents a halide or methosulphate ion.

Optionally, amphoteric surfactants, for example, amine oxides, and zwitterionic surfactants, for example, betaines, may also be present.

Preferably, the quantity of anionic surfactant is in the range of from 5 to 50% by weight of the total composition. More preferably, the quantity of anionic surfactant is in the range of from 8 to 35 wt%, most preferably from 10 to 30 wt%.

Nonionic surfactant, if present, in addition to any which may be present as emulsifier in the speckles, is preferably used in an amount within the range of from 1 to 20 wt% in addition to that which may be present in the structured emulsion.

The total amount of surfactant present is preferably within the range of from 5 to 60 wt%.

The compositions may suitably contain from 10 to 80 wt%, preferably from 15 to 70 wt%, of detergency builder. Preferably, the quantity of builder is in the range of from 15 to 50 wt%.

The detergent compositions may contain as builder a crystalline aluminosilicate, preferably an alkali metal aluminosilicate, more preferably a sodium aluminosilicate (zeolite).

The zeolite used as a builder may be the commercially available zeolite A (zeolite 4A) now widely used in laundry detergent powders. Alternatively, the zeolite may be maximum aluminium zeolite P (zeolite MAP) as described and claimed in EP 384 070B (Unilever), and commercially available as Doucil (Trade Mark) A24 from Crosfield Chemicals Ltd, UK.

Zeolite MAP is defined as an alkali metal aluminosilicate of zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably within the range of from 0.90 to 1.33, preferably within the range of from 0.90 to 1.20.

Especially preferred is zeolite MAP having a silicon to aluminium ratio not exceeding 1.07, more preferably about 1.00. The particle size of the zeolite is not critical. Zeolite A or zeolite MAP of any suitable particle size may be used.

Also preferred according to the present invention are phosphate builders, especially sodium tripolyphosphate.

This may be used in combination with sodium orthophosphate, and/or sodium pyrophosphate.

Other inorganic builders that may be present additionally or alternatively include sodium carbonate, layered silicate, amorphous aluminosilicates.

Most preferably, the builder is selected from sodium tripolyphosphate, zeolite, sodium carbonate, and combinations thereof.

Organic builders may optionally be present. These include polycarboxylate polymers such as polyacrylates and acrylic/maleic copolymers; polyaspartates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-di- and trisuccinates, carboxymethyloxysuccinates, carboxy-methyloxymalonates, dipicolinates, hydroxyethyl iminodiacetates, alkyl- and alkenylmalonates and succinates; and sulphonated fatty acid salts.

Organic builders may be used in minor amounts as supplements to inorganic builders such as phosphates and zeolites. Especially preferred supplementary organic builders are citrates, suitably used in amounts of from 5 to 30 wt%, preferably from 10 to 25 wt%; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, preferably from 1 to 10 wt%.

Builders, both inorganic and organic, are preferably present in alkali metal salt, especially sodium salt, form.

Detergent compositions according to the invention may also suitably contain a bleach system, although non-bleaching formulations are also within the scope of the invention.

The bleach system is preferably based on peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, capable of yielding hydrogen peroxide in aqueous solution. Suitable peroxy bleach compounds include organic peroxides such as urea peroxide, and inorganic persalts such as the alkali metal perborates, percarbonates, perphosphates, persilicates and persulphates. Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate. The peroxy bleach compound is suitably present in an amount of from 5 to 35 wt%, preferably from 10 to 25 wt%.

The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. The bleach precursor is suitably present in an amount of from 1 to 8 wt%, preferably from 2 to 5 wt%.

Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and peroxybenzoic acid precursors; and peroxycarbonic acid precursors. An especially preferred bleach precursor suitable for use in the present invention is N,N,N',N'-tetracetyl ethylenediamine (TAED).

A bleach stabiliser (heavy metal sequestrant) may also be present. Suitable bleach stabilisers include ethylenediamine tetraacetate (EDTA) and the polyphosphonates such as Dequest (Trade Mark), EDTMP.

The detergent compositions may also contain one or more enzymes. Suitable enzymes include the proteases, amylases, cellulases, oxidases, peroxidases and lipases usable for incorporation in detergent compositions.

Preferred proteolytic enzymes (proteases) are catalytically active protein materials which degrade or alter protein types of stains when present as in fabric stains in a hydrolysis reaction. They may be of any suitable origin, such as vegetable, animal, bacterial or yeast origin.

Proteolytic enzymes or proteases of various qualities and origins and having activity in various pH ranges of from 4-12 are available. Proteases of both high and low isoelectric point are suitable.

Other enzymes that may suitably be present include lipases, amylases, and cellulases including high-activity cellulases such as Carezyme (Trade Mark) ex Novo.

In particulate detergent compositions, detergency enzymes are commonly employed in granular form in amounts of from about 0.1 to about 3.0 wt%. However, any suitable physical form of enzyme may be used in any effective amount.

Antiredeposition agents, for example, cellulose esters and ethers, for example sodium carboxymethyl cellulose, may also be present.

The compositions may also contain soil release polymers, for example sulphonated and unsulphonated PET/POET polymers, both end-capped and non-end-capped, and polyethylene glycol/polyvinyl alcohol graft copolymers such as Sokolan (Trade Mark) HP22.

Especially preferred soil release polymers are the sulphonated non-end-capped polyesters described and claimed in WO 95 32997A (Rhodia Chimie).

The detergent compositions may also include one or more inorganic salts other than builder salts. These may include, for example, sodium bicarbonate, sodium silicate, sodium sulphate, magnesium sulphate, calcium sulphate, calcium chloride and sodium chloride. Preferred inorganic salts are sodium sulphate, sodium chloride, and combinations thereof. The coloured speckle composition is especially suitable for incorporation in detergent compositions which contain sodium chloride in addition to that which may be present in the speckle composition.

The detergent compositions may also contain other inorganic materials, for example, calcite, silica, amorphous aluminosilicate, or clays.

Other ingredients that may be present include solvents, hydrotropes, fluorescers, dyes, photobleaches, foam boosters or foam controllers (antifoams) as appropriate, fabric conditioning compounds, and perfumes.

Preparation of the Detergent Composition

Powders of low to moderate bulk density may be prepared by spray-drying a slurry, and optionally postdosing (dry-mixing) further ingredients. "Concentrated" or "compact" powders may be prepared by mixing and granulating processes, for example, using a high-speed mixer/granulator, or other non-tower processes. In both types of powder, the speckle composition may be incorporated by postdosing (dry mixing).

The detergent composition of the invention may alternatively be in tablet form. Tablets may be prepared by compacting powders, especially "concentrated" or "compact" powders, prepared as described above. The speckle composition is then included in the powder prior to compaction.

EXAMPLES

The invention will now be illustrated in further detail by means of the following Examples, in which parts and percentages are by weight unless otherwise stated.

Example 1: Perfumed Pink Speckle Composition

A speckle composition was prepared to the formulation given in Table 1:

Ingredient	wt %
Sodium chloride (d_{50} ~ 1.35 mm)	89.75
Perfume LP 4104 ex Quest	2.25
Ethoxylated alcohol (C_{12} 7EO) (emulsifier)	1.50
Silica	3.00
Zeolite	1.00
Pigment Red CI 12490 and titanium dioxide dispersed in CI 77891, as 5 wt% water suspension	2.00

The composition was prepared as follows. The ingredients were introduced into a stainless steel mixer in the following order:

1. Sodium chloride
2. Silica (1.00 wt%)
3. Coloured perfume emulsion (titanium dioxide, red colourant, perfume, emulsifier, water)

The ingredients were mixed for a period of time sufficient to ensure homogeneity. The resulting material was subsequently layered in the following stages:

4. Silica (1.00 wt%)
5. Zeolite
6. Silica (1.00 wt%)

and the resulting particulate material then dried.

The speckles were of an attractive pink colour and had a pleasant odour.

Example 2: Laundry Detergent Composition

The detergent composition according to Table 2 was made by spray-drying a slurry followed by post-dosing.

33 wt% of the total amount of perfume in the detergent composition is located in the speckles.

During wash tests the detergent composition had a superior perfume release profile.

Table 2

Ingredient	wt %
Spray Dried	
Sodium LAS	14.7
Sodium tripolyphosphate	1.024
Zeolite	4.465
Sodium carbonate	24.22
Sodium chloride	44.37
Sodium carboxymethylcellulose	0.118
Tinopal CBS-x	0.127
Iragon Blue PBG	0.012
Post Dosed	
Speckle of Example 1	4.00
(of which is perfume)	(0.09)
Perfume (not in speckles)	0.18
Sodium sulphate	1.619
Bentonite clay	2.662
Moisture	2.171
Impurities	0.264
Total	**100.0**

Total perfume	0.27%
Perfume in speckles	0.09%
Perfume in bulk powder	0.18%

CLAIMS

1. A perfumed coloured granular composition for use as speckles in a particulate laundry detergent composition characterised in that it comprises at least 1 wt% perfume.

2. A granular composition as claimed in claim 1, characterised in that it comprises at least 1.5 wt% perfume.

3. A granular composition according to claim 1 or claim 2, characterised in that it comprises emulsifier.

4. A granular composition according to claim 3, characterised in that the emulsifier is a C_8 to C_{20}, preferably C_{12} to C_{15}, primary or secondary alcohol ethoxylate ethoxylated with an average of from 1 to 10, preferably 4 to 9, more preferably 5 to 7, moles of ethylene oxide per mole of alcohol.

5. A process for the manufacture of a perfumed coloured granular composition for use as speckles in a particulate laundry detergent composition, characterised in that it comprises the steps of:

   (i) mixing an aqueous perfume emulsion and a colourant with an inorganic granular carrier material; and
   (ii) layering the resultant material with a finely divided porous particulate material.

6. A process according to claim 5, characterised in that the inorganic granular material is layered prior to step (i) with a finely divided porous particulate material.

7. A process according to claims 5 or claim 6, characterised in that the granular carrier material is sodium chloride.

8. A process according to any one of claims 5 to 7, characterised in that the porous particulate layering material comprises at least one material selected from silica and crystalline alkali metal aluminosilicate.

9. A perfumed coloured granular composition obtainable by a process according to any one of claims 5 to 8.

10. A perfumed particulate laundry detergent composition comprising a minor proportion of a coloured speckle composition characterised in that at least 10 wt% of the total amount of perfume in the detergent composition is located in the speckles.

11. A detergent composition according to claim 10, characterised in that at least 20 wt%, preferably at least 30 wt%, of the total amount of perfume is located in the speckles.

12. A detergent composition according to claim 10 or claim 11, characterised in that it comprises a granular composition according to any one of claims 1 to 4 and 9.

13. A detergent composition according to claim 12, characterised in that it comprises from 1 to 10 wt% of the granular composition.

14. A detergent composition according to claim 13, characterised in that it comprises from 2 to 8 wt% of the granular composition.

15. A detergent composition according to any one of claims 10 to 14 characterised in that it comprises from 5 to 60 wt% surfactant and from 10 to 80 wt% builder.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C11D17/00 C11D3/50 C11D3/40 C11D11/00 C11D3/02
C11D3/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DATABASE WPI Section Ch, Week 198620 Derwent Publications Ltd., London, GB; Class A97, AN 1986-128644 XP002222803 & JP 61 066800 A (KAO CORP), 5 April 1986 (1986-04-05) abstract ---	5
X	WO 97 47720 A (ANGELL JOHN WAYNFORTH ;CUTTER GARY RAY (US); PERKIS DAVID FREDERIC) 18 December 1997 (1997-12-18) page 2, line 17 - line 35 page 3, line 8 - line 19 claims 1-10; example I --- -/--	1,2, 10-15

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
28 November 2002	03/01/2003

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Loiselet-Taisne, S

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 02/09051

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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X	US 6 093 258 A (MC LEAN MICHAEL ET AL) 25 July 2000 (2000-07-25) claim 1	1
A	US 5 605 883 A (ILIFF ROBERT J ET AL) 25 February 1997 (1997-02-25) cited in the application column 2, line 27 - line 33 column 6, line 50 - line 62	1-15
P,A	WO 02 10327 A (UNILEVER PLC ;LEVER HINDUSTAN LTD (IN); UNILEVER NV (NL)) 7 February 2002 (2002-02-07) claims; examples	1-15

INTERNATIONAL SEARCH REPORT

Information on patent family members

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